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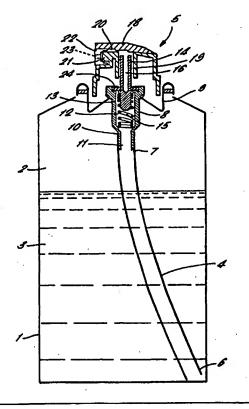
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(54) Title: FRAGRANCE DISPERSION

#### (57) Abstract

A method of improving the dispersion of an airborne active compound, with method comprising directing into a space in which the active compound is to be dispersed liquid droplets from a spray device containing a composition containing an active component, a unipolar charge being imparted to the said liquid droplets by double layer charging during the spraying of the liquid droplets from the spray device, the unipolar charge being at a level such that the said droplets have a charge to mass ratio of at least  $+/-1 \times 10^{-4}$  C/kg.



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#### FRAGRANCE DISPERSION

The present invention relates to a method of dispersing airborne active components and, in particular, to fragrances and insect repellants dispersed from an aerosol spray device.

Dispersion of an active components from an aerosol spray device, such as an aerosol air freshener, is not ideal as the active component generally only has an effect within the direction of the line of spray. This is due to the design requirements for an aerosol spray device which can deliver a spray over a reasonable distance. In particular, the design of the spray head of conventional aerosol spray devices results in the emission of a spray with a small spread angle, that is to say, most of the spray travels at least initially along or close to a central spray line extending from the spray head. Accordingly, if the spray is to be delivered over a considerable spatial volume, including space which has a substantial lateral radiation having regard to its centerline, a large amount of aerosol spray must be delivered in order to ensure that the active component, such as a fragrance or insect repellant, reaches throughout the target space.

We have now developed a method whereby active components, such as fragrances or insect repellants, may be more effectively distributed throughout a particular space.

According to the present invention there is

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provided a method of improving the dispersion of an airborne active component, which method comprises directing into a space in which the active component is to be dispersed liquid droplets from a spray device containing a composition containing the active component, a unipolar charge being imparted to the said liquid droplets by double layer charging during the spraying of the liquid droplets from the spray device, the unipolar charge being at a level such that the said droplets have a charge to mass ratio of at least +/- 1 x 10<sup>-4</sup> C/kg.

The composition which is dispersed according to the method of the invention may be a fragrance composition or an insect repellant.

The fragrance composition which is dispersed according to the present invention contains at least one fragrance component. Examples of such fragrance components are diethylphthalate, orange terpenes (limonene), styrallyl acetate ester, Cyclacet, methyl ionone ketone, vanillin, Litsea Cybeba, 2-phenylethan-1-ol, dipropylene glycol and  $\alpha$ -methyl-p-3°-butyl hydrocinnamic aldehyde.

It is preferred that the unipolar charge which is imparted to the liquid droplets is generated solely by the interaction between the liquid within the spray device and the spray device itself as the liquid is sprayed therefrom. In particular, it is preferred that the manner in which a unipolar charge is imparted to the liquid droplets does not rely even partly upon the connection of the spray device to any external charge including device, such as a source of

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relatively high voltage, or any internal charge inducing device, such as a battery. With such an arrangement, the spray device is entirely self-contained making it suitable for use both in industrial, institutional and domestic situations.

Preferably, the spray device is a domestic pressure-spraying device devoid of any electrical circuitry but which is capable of being hand held. Typically such a device has a capacity in the range of from 10ml to 2000ml and can be actuated by hand, or by an automatic actuating mechanism. A particularly preferred domestic device is a hand-held aerosol can.

Preferably, therefore the droplet charge to mass ratio of at least +/- 1 x 10<sup>-4</sup> C/kg is imparted to the liquid droplets as a result of the use of an aerosol spray device with at least one of the features of the material of the actuator, the size and shape of the orifice of the actuator, the diameter of the dip tube, the characteristics of the valve and the formulation of the fragrant composition containers within the aerosol spray device being chosen in order to achieve the said droplet charge to mass ratio by double layer charging imparting the unipolar charge to the droplets during the actual spraying of the liquid droplets from the orifice of the aerosol spray device.

As a result of the method of the present invention, a volume of space to be treated, such as the interior of a room, may be quickly and effectively filled with the active component using a much smaller amount of an aerosol composition, such as a room spray, than has occurred hitherto. Accordingly, more

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effective fragrancing of a room can be obtained using a fragrance composition, and more effective dispersion of an insect repellant using an insect repellant composition.

This result is achieved because of the unipolar charge imparted to the liquid droplets of the aerosol spray. This charge has two effects. Since all of the droplets carry the same polarity charge, they are repelled one from another. Accordingly, there is little or no coalescence of the droplets and, in contrast, they tend to spread out to a great extent as compared to uncharged droplets. In addition, if the repulsive forces from the charge within the droplets is greater than the surface tension force of the droplets, the droplets are caused to fragment into a plurality of smaller charged droplets (exceeding the Rayleigh limit). This process continues until either the two opposing forces are equalised or the droplet has fully evaporated.

The liquid composition which is sprayed into the air using the aerosol spray device is preferably a water and hydrocarbon mixture, or emulsion, or a liquid which is converted into an emulsion by shaking the spraying device before use, or during the spraying process.

Whilst all liquid aerosols are known to carry a net negative or positive charge as a result of double layer charging, or the fragmentation of liquid droplets, the charge imparted to droplets of liquid sprayed from standard devices is only of the order of  $\pm 1 \times 10^{-8}$  to  $1 \times 10^{-5}$  C/kg.

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The invention relies on combining various characteristics of the design of an aerosol spray so as to increase the charging of the liquid as it is sprayed from the aerosol spray device.

A typical aerosol spray device comprises:

- An aerosol can containing the composition to be sprayed from the device and a liquid or gaseous propellant;
- 2. A dip tube extending into the can, the upper end of the dip tube being connected to the valve;
- 3. An actuator situated above the valve which is capable of being depressed in order to operate the valve; and
- An insert provided in the actuator comprising an orifice from which the composition is sprayed.

A preferred aerosol spray device for use in the present invention is described in WO 97/12227.

It is possible to impart higher charges to the liquid droplets by choosing aspects of the aerosol device including the material, shape and dimensions of the actuator, the actuator insert, the valve and the dip tube and the characteristics of the liquid which is to be sprayed, so that the required level of charge is generated as the liquid is dispersed as droplets.

A number of characteristics of the aerosol system increase double layer charging and charge exchange between the liquid formulation and the surfaces of the aerosol system. Such increases are brought about by factors which may increase the turbulence of the flow

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through the system, and increase the frequency and velocity of contact between the liquid and the internal surfaces of the container and valve and actuator system.

By way of example, characteristics of the actuator can be optimised to increase the charge levels on the liquid sprayed from the container. A smaller orifice in the actuator insert, of a size of 0.45mm or less, increases the charge levels of the liquid sprayed through the actuator. The choice of material for the actuator can also increase the charge levels on the liquid sprayed from the device with material such as nylon, polyester, acetal, PVC and polypropylene tending to increase the charge levels. The geometry of the orifice in the insert can be optimised to increase the levels on the liquid as it is sprayed through the actuator. Inserts which promote the mechanical break-up of the liquid give better charging.

The actuator insert of the spray device may be formed from a conducting, insulating, semi-conducting or static-dissipative material.

The characteristics of the dip tube can be optimised to increase charge levels in the liquid sprayed from the container. A narrow dip tube, of for example about 1.27mm internal diameter, increases the charge levels on the liquid, and the dip tube material can also be changed to increase charge.

Valve characteristics can be selected which increase the charge to mass ratio of the liquid product as it is sprayed from the container. A small

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tailpiece orifice in the housing, of about 0.65mm, increases product charge to mass ratio during spraying. A reduced number of holes in the stem, for example 2 x 0.50mm, also increases product charge during spray. The presence of a vapour phase tap helps to maximise the charge levels, a large orifice vapour phase tap of, for example, about 0.50mm to 1.0mm generally giving higher charge levels.

Changes in the product formation can also affect charging levels. A formulation containing a mixture of hydrocarbon and water, or an emulsion of an immiscible hydrocarbon and water, will carry a higher charge to mass ratio when sprayed from the aerosol device than either a water alone or hydrocarbon alone formulation.

It is preferred that an air freshener composition of use in the present invention comprises an oil phase, an aqueous phase, a surfactant, a fragrance component and a propellant.

Preferably the oil phase includes a  $C_9$  -  $C_{12}$  hydrocarbon which is preferably present in the composition in the amount of from 2 to 10% w/w.

Preferably the surfactant is glyceryl oleate or a polyglycerol oleate, preferably present in the composition in an amount of from 0.1 to 1.0% w/w.

preferably the propellant is liquified petroleum gas (LPG) which is preferably butane, optionally in admixture with propane. The propellant may be present in an amount of from 10 to 90% w/w depending upon whether the composition is intended for spraying as a "wet" or as a "dry" composition. For a "wet"

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composition, the propellant is preferably present in an amount of from 20 to 50% w/w, more preferably in an amount of from 30 to 40% w/w.

The liquid droplets sprayed from the aerosol spray device will generally have diameters in the range of from 5 to 100 micrometres, with a peak of droplets of about 40 micrometres. The liquid which is sprayed from the aerosol spray device may contain a predetermined amount of a particulate material, for example, fumed silica, or a predetermined amount of a volatile solid material, such as menthol or naphthalene.

A can for a typical aerosol spray device is formed of aluminum or lacquered or unlacquered tin plate or the like. The actuator insert may be formed or, for instance, acetal resin. The valve stem lateral opening may typically be in the form of two apertures of diameters 0.51mm.

The present invention will now be described, by way of example only, with reference to the accompanying drawings, in which:-

Figure 1 is a diagrammatic cross section through an aerosol spraying apparatus in accordance with the invention:

Figure 2 is a diagrammatic cross section though the valve assembly of the apparatus of Figure 1;

Figure 3 is a cross section through the actuator insert of the assembly shown in Figure 2;

Figure 4 shows the configuration of the bore of the spraying head shown in Figure 3 when viewed in the direction A;

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Figure 5 shows the configuration of the swirl chamber of the spraying head shown in Figure 3 when viewed in the direction B; and

Figures 6 to 10 illustrate tests and results using methods in accordance with the present invention.

Referring to Figures 1 and 2, an aerosol spray device in accordance with the invention is shown. It comprises a can 1, formed of aluminum or lacquered or unlacquered tin plate or the like in conventional manner, defining a reservoir 2 for a liquid 3 having a conductivity such that droplets of the liquid can carry an appropriate electrostatic charge. Also located in the can is a gas under pressure which is capable of forcing the liquid 3 out of the can 1 via a conduit system comprising a dip tube 4 and a valve and actuator assembly 5. The dip tube 4 includes one end 6 which terminates at a bottom peripheral part of the can 1 and another end 7 which is connected to a tailpiece 8 of the valve assembly. The tailpiece 8 is secured by a mounting assembly 9 fitted in an opening in the top of the can and includes a lower portion 10 defining a tailpiece orifice 11 to which end 7 of the dip tube 4 is connected. The tailpiece includes a bore 12 of relatively narrow diameter at lower portion 11 and a relatively wider diameter at its upper portion 13. The valve assembly also includes a stem pipe 14 mounted within the bore 12 of the tailpiece and arranged to be axially displaced within the bore 12 against the action of spring 15. The valve stem 14 includes an internal bore 16 having one or more

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lateral openings (stem holes) 17 (see Figure 2). The valve assembly includes an actuator 18 having a central bore 19 which accommodates the valve stem 14 such that the bore 16 of the stem pipe 14 is in communication with bore 19 of the actuator. A passage 20 in the actuator extending perpendicularly to the bore 19 links the bore 19 with a recess including a post 21 on which is mounted a spraying head in the form of an insert 22 including a bore 23 which is in communication with the passage 20.

A ring 24 of elastomeric material is provided between the outer surface of the valve stem 14 and, ordinarily, this sealing ring closes the lateral opening 17 in the valve stem 14. The construction of the valve assembly is such that when the actuator 18 is manually depressed, it urges the valve stem 14 downwards against the action of the spring 15 as shown in Figure 2 so that the sealing ring 24 no longer closes the lateral opening 17. In this position, a path is provided from the reservoir 2 to the bore 23 of the spraying head so that liquid can be forced, under the pressure of the gas in the can, to the spraying head via a conduit system comprising the dip tube 4, the tailpiece bore 12, the valve stem bore 16, the actuator bore 19 and the passage 20.

An orifice 27 (not shown in Figure 1) is provided in the wall of the tailpiece 8 and constitutes a vapour phase tap whereby the gas pressure in the reservoir 2 can act directly on the liquid flowing through the valve assembly. This increases the turbulence of the liquid. It has been found that an

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increased charge is provided if the diameter of the orifice 27 is at least 0.76mm.

Preferably the lateral opening 17 linking the valve stem bore 16 to the tailpiece bore 12 in the form of 2 orifices each having a diameter of not more than 0.51mm to enhance electrostatic charge generation. Further, the diameter of the dip tube 4 is preferably as small as possible, for example, 1.2mm, in order to increase the charge imparted to the liquid. Also, in charge generation is enhanced if the diameter of the tailpiece orifice 11 is as small as possible eg not more than about 0.64mm.

Referring now to Figure 3, there is shown on an increased scale, a cross section through the actuator insert of the apparatus of Figures 1 and 2. For simplicity, the bore 23 is shown as a single cylindrical aperture in this Figure. However, the bore 23 preferably has the configuration, for instance, shown in Figure 4. The apertures of the bore 23 are denoted by reference numeral 31 and the aperture-defining portions of the bore are denoted by reference numeral 30. The total peripheral length of the aperture-defining portions at the bore outlet is denoted by L(in mm) and a is the total area of the aperture at the bore outlet (in mm<sup>2</sup>) and the values for L a are as indicated in Figure 4. L/a exceeds 8 and this condition has been found to be particularly conductive to charge development because it signifies an increased contact area between the actuator insert and the liquid passing there through.

Many different configurations can be adopted in

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order to produce a high L/a ratio without the crosssectional area a being reduced to a value which would allow only low liquid flow rates. Thus, for example it is possible to use actuator insert bore configurations (i) wherein the bore outlet comprises a 5. plurality of segment-like apertures (with or without a central aperture); (ii) wherein the outlet comprises a plurality of sector-like apertures; (iii) wherein the aperture together form an outlet in the form of a grill or grid; (iv) wherein the outlet is generally 10 cruciform; (v) wherein the apertures together define an outlet in the form of concentric rings; and combinations of these configurations. Particularly preferred are actuator insert bore configurations 15 wherein a tongue like portion protrudes into the liquid flow stream and can be vibrated thereby. vibrational property may cause turbulent flow and enhanced electrostatic charge separation of the double layer allowing more charge to move into the bulk of 20 the liquid.

Referring now to Figure 5, there is shown a plan view of one possible configuration of swirl chamber 35 of the actuator insert 22. The swirl chamber includes 4 lateral channels 36 equally spaced and tangential to a central area 37 surrounding the bore 23. In use, the liquid driven from the reservoir 2 by the gas under pressure travels along passage 20 and strikes the channels 36 normal to the longitudinal axis of the channels. The arrangement of the channels is such that the liquid tends to follow a circular motion prior to entering the central area 37 and thence the

bore 23. As a consequence, the liquid is subjected to substantial turbulence which enhances the electrostatic charge in the liquid.

The following Examples illustrate the invention:-

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#### EXAMPLE 1

An air freshener composition was prepared from the following components:

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	% w/w
Mono propylene glycol	0.3
Sorbitan mono oleate	0.85
Fragrance	0.85
Water	63
Liquified petroleum gas	35

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The composition was introduced into a tinplate aerosol can having valve assemblies comprising 3mm polyethylene dip tube 4, 0.64mm tailpiece orifice 11, 1.2mm vapour phase tap 27 and 4 x 0.61mm valve stem lateral openings 17. The actuator 18 was an Accusol type fitted with a 0.46mm MBU CO<sub>2</sub> actuator insert 22 (both supplied by Precision Valve).

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On depression of the actuator 18, a fine spray of liquid droplets having a charge/mass value of -1.7 x 10 C/kg and a flow rate of approximately 0.4g per second was obtained. The droplets became rapidly dispersed in the air.

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The above described aerosol spray device was

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compared with a standard, known aerosol device loaded with the same formulation, having a charge/mass value of  $-5 \times 10^6$  C/kg and a flow rate of 1g per second being obtained.

When equal masses of each product were sprayed to disperse fragrance in a room, it was found that the spray of the present invention resulted in the fragrance being more evenly dispersed throughout the room with a perceived higher level of fragrance, which lasted longer as compared to the standard known device.

#### EXAMPLE 2

An example of a basic formulation for an aerosol composition of use in the method of the present invention is as follows:

	% w/w
Butane 40 propellant	35
Hydrocarbon, eg Isopar G	5
Surfactant, eg Polyglycerol Oleate	0.3
Corrosion Inhibitor, e.g. Butylated Hydroxy Toluene	0.013
Fragrance	0.7
Soft Water (to make up to 100%)	58.987

EXAMPLE 3 - Comparative Tests

Fragrance Speed and Duration Measurement

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Experiments were devised in order to test the method of the present invention against a similar method in which the liquid droplets are supplied from a standard aerosol spray device and have a charge mass ratio of less than +/- 1 x  $10^{-4}$  C/kg.

The experiment was panel based. In order to be able to assess the results, it was necessary to measure the threshold levels of the panellists. By choosing panellists of the same threshold value, a more accurate result could be obtained.

#### THRESHOLD TESTING

6 Samples of fragrance with diethyl phthalate (DEP) were made up at different fragrance concentrations. They were placed in amber, wide necked glass jars and labeled as follows:

A = 2% solution, 0.6g fragrance diluted with 29.4g DEP

B = 0.5% solution, 0.15g fragrance diluted with 29.85g DEP

C = 0.05% solution, 0.015g fragrance diluted with 29.985g DEP

D = 0.005% solution, 0.0015g fragrance diluted with 29.9985g DEP

E = 0.0005% solution, 0.00015g fragrance diluted with 29.99985g DEP

F = Standard, 30g DEP

G = Standard, 30g DEP

A group of 10 prospective panellists were then

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asked to pick "the odd one out" using F + G as the standard samples all the time and any one of the other samples. Starting at the strongest, (A), they were asked three times to pick out the samples they suspected to be fragranced.

The panellists who achieved consistent correctly answers for fragrances in the ranges A - C were chosen as final panellists for the following experiment. It should be noted that none of the panellists were able to predict fragrance D with 100% certainty.

#### SPEED OF TRAVEL AND DURATION MEASUREMENT

Six people were chosen from the panel tested above to perform the testing. These same people were used throughout the test. The selection of the panellists was based on them having similar threshold levels to one other.

The panellists were positioned at various locations within the enclosed area. The same enclosed area was used throughout the tests being conducted. The panellists wore blindfolds and ear defenders. Music was played so that they were unaware of any noise produced by spraying the aerosols.

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The panellists were placed at random positions throughout the room (see Figure 6), and the door to the room was closed. One of the test initiators started a stop-clock at the same time as another sprayed a measured amount of the following air freshener formulation into the room.

	% w/w
Propylene Glycol	0.3
Sorbitol Oleate	0.85
Perfume	0.85
Soft Water	62.88
Sodium Nitrite	0.12
Butane 40	35
TOTAL	100

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The panellists were asked to raise their hands for 5 seconds when they first smelt the fragrance. The time taken for each individual to detect the fragrance from its dispensing time was noted. The panellists were asked to sit down when they could no longer smell the fragrance. This time was also noted. If after 10 minutes from the initial spray time, any panellists were still standing, the test was discontinued and the remaining panellists were asked whether they thought the fragrance level had changed from the time that they first smelt it.

Each test was repeated in order to obtain statistically significant results.

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#### Results

The results from the tests are given in Figures 7 and 8 and Tables 1 and 2. The term "Charged Aerosol" denotes an electrostatically charged aerosol spray device as described above and which, with the

particular formulation, produces liquid droplets with a charge to mass ratio of at least  $+/-1 \times 10^{-4}$  C/kg. The speed of travel of the fragrance is enhanced and the duration of the fragrance is dramatically increased.

Position	Standard	Charged	
•	Time Sec	Time (sec)	
Position 1	37.25	24.5	
Position 2	39.56	25.36	
Position 3	46.13	36.36	
Position 4	54.56	63.67	
Position 5	62.38	61.3	
Position 6	66	66.33	
Position 7	43	32.67	

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Table 1 speed of Travel of the Fragrance to the Various Positions

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Position	Standard	Charged	
	Time Sec	Time (sec)	
Position 1	92	152.5	
Position 2	109.89	195.09	
Position 3	107.13	179.36	
Position 4	137	313.56	
Position 5	104.57	352.1	
Position 6	123.22	309.33	
Position 7	116.5	223.33	

Table 2 Duration of the Fragrance to the Various Positions

### FRAGRANCE DISTRIBUTION TESTING

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Again this experiment was panel based, the six panellists selected had a common fragrance threshold level and were used throughout the series of tests.

An enclosed area was selected and marked as in Figure 9. The aerosol compositions which were compared were as described above in this Example. The premarked area was sprayed with the aerosol to be tested with a measured amount of spray. The door was then sealed. Three minutes after the spray was dispensed, the panellists moved very slowly into the room. The rate of movement was kept to a minimum to reduce any air disturbances to the room. This protocol was repeated throughout the test session.

The panellists wore respirator masks. Each panellist moved to the premarked areas in the room by crawling and then standing up at the spot. At this point each of them removed their masks and smelt the air, noting the fragrance strength in that area. They noted the strength on standard strength test sheets they had been provided with. They then replaced their respirators and once again crawled to the next point from 1-10. The panellists always followed the same order of points, and moved in the same panellist order from 1 to 6.

The results are given in Figure 10. Clearly, there is a consistent increase in the perceived

strength of the fragrance in the locations tested for the composition sprayed according to the method of the invention.

#### EXAMPLE 4

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An example of a basic formulation for an insect repellant composition of use in the method of the present invention is as follows:

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	% w/w
Butane 40 propellant	35
Hydrocarbon, eg Isopar G	5
Surfactant, eg Polyglycerol Oleate	0.3
Corrosion Inhibitor, e.g.	0.013
Butylated Hydroxy Toluene	
Citronellal	0.7
Soft Water (to make up to 100%)	58.987

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#### CLAIMS:

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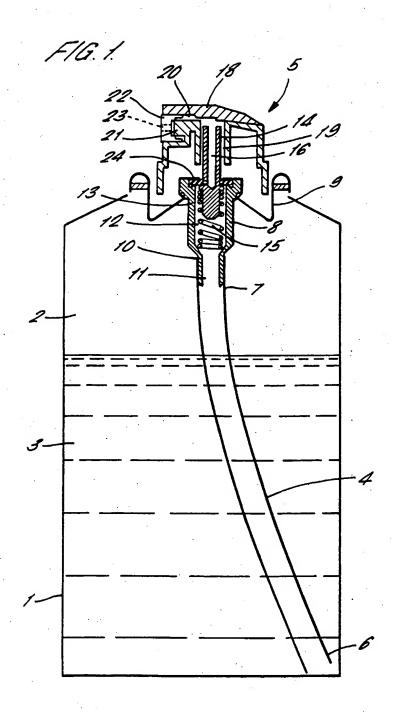
- 1. A method of improving the dispersion of an airborne active compound, with method comprising directing into a space in which the active compound is to be dispersed liquid droplets from a spray device containing a composition containing an active component, a unipolar charge being imparted to the said liquid droplets by double layer charging during the spraying of the liquid droplets from the spray device, the unipolar charge being at a level such that the said droplets have a charge to mass ratio of at least +/- 1 x 10<sup>-4</sup> C/kg.
- 2. A method as claimed in claim 1 wherein the spray device is an aerosol spray device.
  - 3. A method as claimed in claim 1 or claim 2 wherein the composition contained in the aerosol spray device is an emulsion.
  - 4. A method as claimed in any one of the preceding claims wherein the liquid droplets have a diameter in the range of from 5 to 100 micrometres.
  - 5. A method as claimed in any one of the preceding claims wherein the composition containing an active component is a fragrant composition or an insect repellant.
    - 6. A method as claimed in claim 5 wherein the

fragrant composition includes a fragrance component selected from one or more of diethylphthalate, orange terpenes (limonene), styrallyl acetate ester, Cyclacet, methyl ionone ketone, vanillin, Litsea Cybeba, 2-phenylethan-1-ol, dipropylene glycol and  $\alpha$ -methyl- $\rho$ -3°-butyl hydrocinnamic aldehyde.

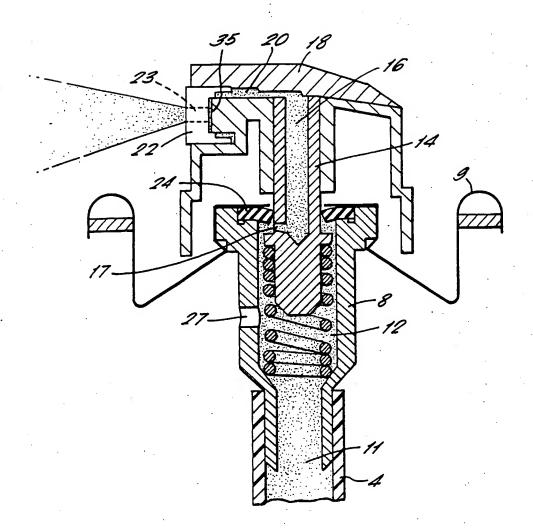
- 7. A method as claimed in any one of the preceding claims wherein the unipolar charge is imparted to the liquid droplets solely by the interaction between the liquid and the spray device, without any charge being imparted thereto from an internal or external charge inducing device.
- A method as claimed in any one of the 15 preceding claims wherein the droplet charge to mass ratio of at least  $+/-1 \times 10^{-4}$  C/kg is imparted to the liquid droplets as a result of the use of an aerosol spray device with at least one of the features of the material of the actuator, the size and shape of the 20 orifice of the actuator, the diameter of the dip tube, the characteristics of the valve and the formulation of the composition contained within the aerosol spray device being chosen in order to achieve the said droplet charge to mass ratio by double layer charging 25 imparting the unipolar charge to the droplets during the actual spraying of the liquid droplets from the orifice of the aerosol spray device.
- 9. A method as claimed in any cne of the preceding claims wherein the composition comprises an

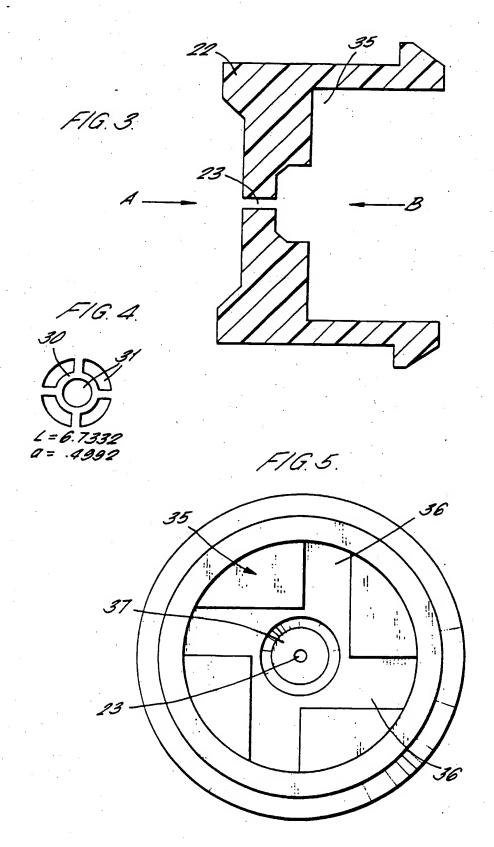
oil phase, an aqueous phase, a surfactant, a fragrance and a propellant.

- 10. A method as claimed in claim 9 wherein the oil phase includes a C, C<sub>12</sub> hydrocarbon.
  - 11. A method as claimed in claim 10 wherein the  $C_9$   $C_{12}$  hydrocarbon is present at the composition in an amount of from 2 to 10% w/w.
  - 12. A method as claimed in any one of claims 9 to 11 wherein the surfactant is glyceryl oleate or a polyglycerol oleate.
- 13. A method as claimed in any one of claims 9 to 12 wherein the surfactant is present in the composition in an amount of from 0.1 to 1.0% w/w.
- 14. A method as claimed in any one of claims 9 20 to 13 wherein the propellant is liquified petroleum gas.
- 15. A method as claimed in claim 14 wherein the propellant is present in the composition in an amount of from 20 to 50% w/w.



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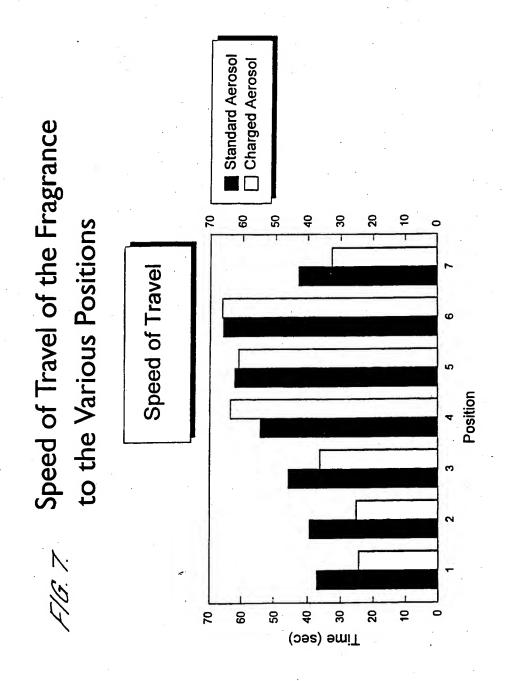


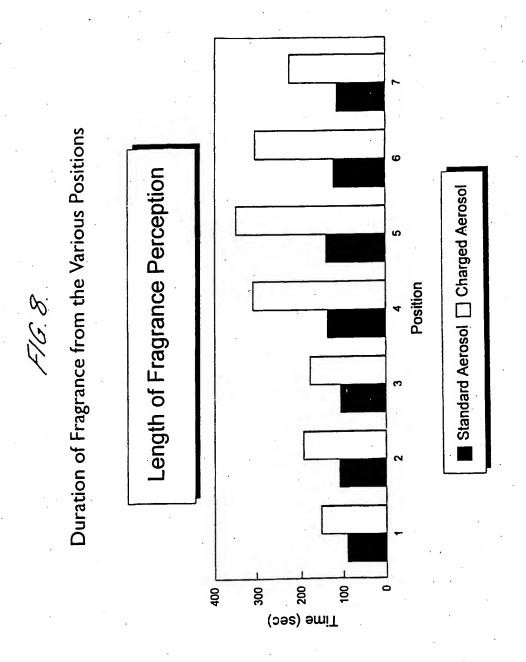


F/G. 6.

Plan of Room to Illustrate Test

X Position 4	X Position 5	X Position 6
X Position 3		X Position 7
X Position 2	·	**
X Position I		
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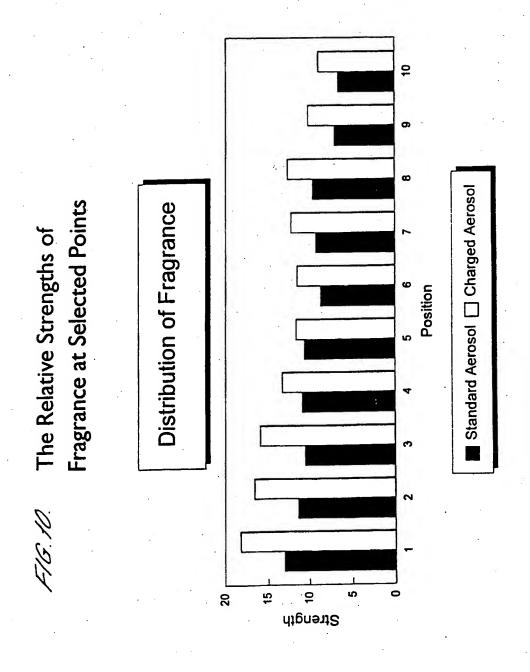




F1G. 9.

Positioning in the Room of the Fragrance Strength Test Areas.

	I X	3 X	7 X		
4 X		90		2 X	
8 X		**			
10 X	5 X	Direction of Spray	of		6 X



### INTERNATIONAL SEARCH REPORT

Inter 'enal Application No PCT/GB 99/01973

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 A61L9/14

According to International Patent Classification (IPC) or to both national classification and IPC

#### B. FIELDS SEARCHED

 $\begin{array}{lll} \mbox{Minimum documentation searched (classification system followed by classification symbols)} \\ \mbox{IPC 7} & \mbox{A61L} & \mbox{B01D} & \mbox{B05B} & \mbox{B08B} \\ \end{array}$ 

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical search terms used)

Category ?	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 97 28883 A (FOX RODNEY THOMAS ; HARRISON NEALE MARK (GB); HUGHES JOHN FARRELL () 14 August 1997 (1997-08-14) page 1, line 23 - line 29 page 2, line 27 - line 35 page 3, line 3 - line 34	1-5, 7-10,14
X	WO 98 24356 A (GAYNOR PAUL TERENCE ;HUGHES JOHN FARRELL (GB); MCKECHNIE MALCOLM T) 11 June 1998 (1998-06-11) page 2, line 29 - line 34 page 5, line 6 - line 13 page 9, line 30 - line 35	1,2,4,7,
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Further documents are listed in the continuation of box C.	Patent family members are listed in annex.
3 Special categories of cited documents:  A document defining the general state of the art which is not considered to be of particular relevance  E earlier document but published on or after the international	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention.  "X" document of particular relevance; the claimed invention.
tiling date  "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the
"O" document referring to an oral disclosure, use. exhibition or other means  "O" document published prior to the international filling date but later than the priority date claimed	document is combined with one or more other such docu- ments, such combination being obvious to a person skilled in the art.  "&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report
21 September 1999	30/09/1999
Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5618 Patentiaan 2 NL – 2260 HV Rijswijk Tel. (+31-70) 340–2040, Tx, 31 651 epo nl. Fax: (+31-70) 340–3016	Menidjel, R

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# INTERNATIONAL SEARCH REPORT

Inter Ional Application No PC1/GB 99/01973

C.(Continua Category *	ition) DOCUMENTS CONSIDERED T BE RELEVANT  Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Α .	US 4 776 515 A (MICHALCHIK MICHAEL) 11 October 1988 (1988-10-11) column 2, line 57 - line 61 column 3, line 11 - line 14 column 4, line 3 - line 8	1-3,5,8
A	EP 0 572 080 A (QUEST INT) 1 December 1993 (1993-12-01) page 5, line 35 - line 48 page 6, line 20 - line 39	1-3,5,6, 9-13
A	US 5 085 849 A (SAMPSON DENNIS ET AL) 4 February 1992 (1992-02-04) column 1, line 60 -column 2, line 23 claims 1,4-6,8,9	1,3,5,6, 9,14,15
	*	
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1

### INTERNATIONAL SEARCH REPORT

Transition on patent family members

PCT/GB 99/01973

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
WO 9728883	Α	A 14-08-1997	AU	1613397 A	28-08-1997
			BR	9707383 A	13-04-1999
·.	•		CN	1214640 A	21-04-1999
			EP	0888162 A	07-01-1999
		. 1	NZ	331255 A	30-08-1999
WO 9824356	A	11-06-1998	GB	2328862 A	10-03-1999
			AU	5401798 A	29-06-1998
US 4776515	A	11-10-1988	NONE		
EP 0572080	Α	01-12-1993	EP	0571677 A	01-12-1993
			AT	130182 T	15-12-1995
			DE	69300796 D	21-12-1995
			DE	69300796 T	18-04-1996
		•	JP	6040877 A	15-02-1994
			US	5374614 A	20-12-1994
US 5085849	Α	04-02-1992	NONE		

Form PCT/ISA/210 (patent family annex) (July 1992)